*भारतीय मानक*
**IS 15080 : 2022**

***Indian Standard***

# 6-ब्रोमो-2,4-डाईनाइट्रोएनीलीन, तकनीकी — विशिष्टि

*( पहला पुनरीक्षण )*

# 6-Bromo-2,4-dinitroaniline,Technical — Specification

*( First Revision )*

ICS 71.080.30

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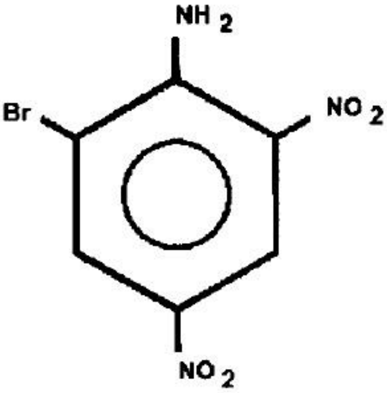
**October 2022** **Price Group 6**

Dye Intermediates Sectional Committee, PCD 26

## FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

6-Bromo-2,4-dinitroaniline ($C_6H_4O_4N_3Br$) is an important intermediate, used in manufacture of disperse dyes represented by following structural formula:



6-BROMO-2,4-DINITROANILINE

[MOLECULAR MASS: 262]

(CAS REGISTRY NO.:1817-73-8)

This Standard was first published in 2001. The Committee responsible for the preparation of this standard decided to revise it in order to incorporate recent development in analytical techniques and use of more sophisticated instrument to determine purity and impurity profile. In this (first) revision test methods for determination by purity and impurity of 2,4-dinitroaniline content are updated by substituting gas chromatographic method by high performance liquid chromatography (HPLC) method.

The composition of the Committee responsible for formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Indian Standard*

# 6-BROMO-2,4-DINITROANILINE, TECHNICAL — SPECIFICATION

*(First Revision)*

## 1 SCOPE

This standard prescribes the requirements, methods of sampling and testing for 6-Bromo-2,4-dinitroaniline.

## 2 REFERENCES

The following standards contain provisions, which through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards given below:

| *IS No.* | *Title* |
|---|---|
| 1070 : 1992 | Reagent grade water — Specification *(third revision)* |
| 2552 : 1989 | Steel drums (galvanized and ungalvanized) — Specification (*third revision*) |
| 5299 : 2001 | Methods of sampling and tests for dye intermediates *(first revision)* |

## 3 REQUIREMENTS

### 3.1 Description

The material shall be orange to brownish yellow dry powder and shall be free from visible impurities.

**3.2** The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in col 4 and col 5 of Table 1.

**Table 1 Requirements for 6-Bromo-2,4-dinitroaniline**

(*Clauses* 3.2, 5.3.1, 5.3.2 *and* 6.1)

| **Sl No.** | **Characteristic** | **Requirement** | **Method of Test, Ref** | |
|---|---|---|---|---|
| | | | Annex | Clause No. in IS 5299 |
| (1) | (2) | (3) | (4) | (5) |
| i) | Assay (by HPLC), percent by mass, *Min* | 98.0 | A | — |
| ii) | Melting Point, °C, *Min* | 148 | — | **9** |
| iii) | Matter insoluble in methanol, percent by mass, *Max* | 0.2 | — | **11** |
| iv) | 2,4-dinitroaniline content, percent by mass, *Max* | 1.0 | B | — |

## 4 PACKING AND MARKING

### 4.1 Packing

The material shall be packed in suitable paper/plastic bags, plywood boxes or drums (*conforming to* IS 2552).

### 4.2 Marking

**4.2.1** Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of manufacturer and his recognized trade-mark, if any;

c) Net mass of material;

d) Month and year of the manufacture;

e) Batch or lot number; and

f) Any other statutory requirements.

**4.2.2** *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

## 5 SAMPLING

**5.1** The method of drawing representative samples of the material shall be as prescribed in **4** of IS 5299.

### 5.2 Numbers of Tests

**5.2.1** Tests for description, determination of melting point, 2,4-dinitroaniline and assay shall be conducted on the individual samples.

**5.2.2** Tests for the determination of the remaining characteristic, that is, matter insoluble in methanol shall be performed on the composite sample.

### 5.3 Criteria for Conformity

**5.3.1** *For Individual Samples*

The lot shall be declared as conforming to the requirements of the description, melting point,2,4-dinitroaniline content and assay, if each of the individual test results satisfies the relevant requirements given at **3.1** and in Table 1.

**5.3.2** *For Composite Sample*

For declaring the conformity of the lot to matter insoluble in methanol tested on the composite sample, the test result shall satisfy the relevant requirement given in Table 1.

## 6 TEST METHODS

**6.1** Tests shall be conducted according to the methods prescribed and as indicated in col 4 and col 5 of Table 1.

### 6.2 Quality of Reagents

Unless specified otherwise, ‘pure chemicals’ and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the results of analysis.

## ANNEX A

[*Table 1, S1 No.* (i)]

## DETERMINATION OF 6-BROMO-2,4-DINITROANILINE CONTENT (ASSAY) BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

### A-1 GENERAL

High-performance liquid chromatography or high-pressure liquid chromatography (HPLC) is a chromatographic method that is used to separate a mixture of compounds in analytical chemistry and biochemistry so as to identify, quantify or purify the individual components of the mixture.

### A-2 APPARATUS

**A-2.1 Binary Gradient Liquid Chromatography System**, capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

**A-2.2 Column**

C18 column of 100 Å with length 250 m, internal diameter 4.6 mm and particle size 5 µm or equivalent.

### A-3 REAGENTS

**A-3.1 Acetonitrile**, HPLC grade.

**A-3.2 Water**, HPLC Grade.

**A-3.3 Tetra-*n*-butylammonium Hydrogen Sulphate**

**A-3.4 6-Bromo-2,4-dinitroaniline**, known purity.

### A-4 STANDARD PREPARATION

Weigh accurately 0.050 0 g standard 6-bromo-2,4-dinitroaniline in 100 ml volumetric flask. Dissolve it in acetonitrile and make it up to the mark with acetonitrile.

### A-5 SAMPLE PREPARATION

Weigh accurately 0.050 0 g sample in 100 ml volumetric flask. Dissolve it in acetonitrileand make it upto the mark with acetonitrile.

### A-6 BUFFER PREPARATION

Take 1.00 g tetra-*n*-butylammonium hydrogen sulphate in 500 ml volumetric flask. Add 200 ml HPLC grade water and completely dissolve it. Make it up to the mark with HPLC grade water.

**A-7 FLOW RATE,** 1.00 ml/min.

### A-8 MOBILE PHASE

| Acetonitrile | Buffer |
|---|---|
| 40 | 60 |

**A-9 COLUMN OVEN TEMPERATURE,** 40 °C.

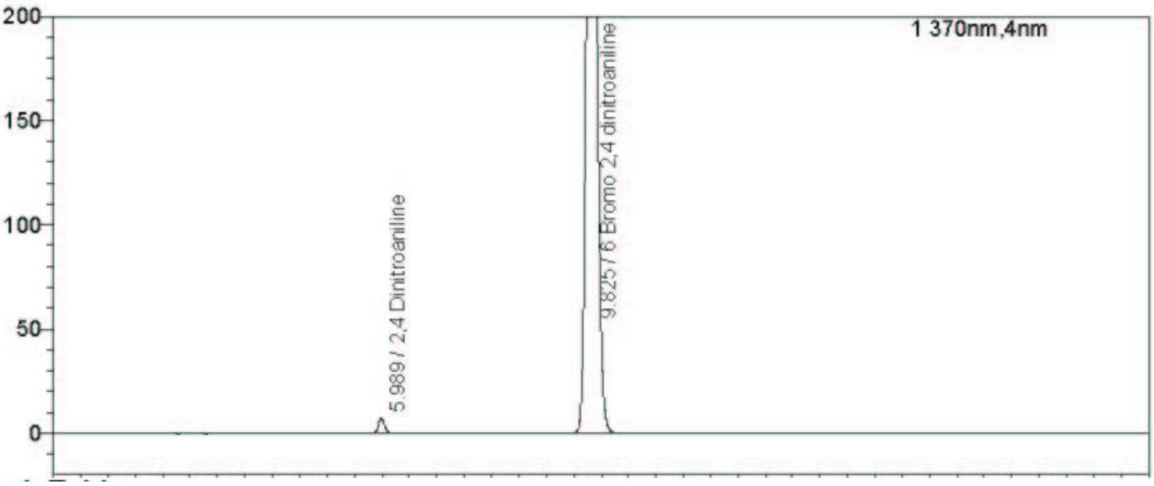
**A-10 INJECTION VOLUME,** 5 µl.

**A-11 RUN TIME,** 20 min.

**A-12 WAVELENGTH,** 370 nm.

### A-13 PEAK TIME

6-bromo-2,4-dinitroaniline: 9.28 min



## A-14 CALCULATION

Calculate the peak area of individual constituent pertaining to 6-bromo-2,4-dinitroaniline on the chromatogram of the material. The concentration of the constituent may be obtained on the basis ofpeak area on chromatogram obtained with known amount of pure 6-bromo-2,4-dinitroaniline.

$$6-\text{bromo}-2{,}4-\text{dinitroaniline, percent}=\frac{A_2\ \text{x}\ V_1\ \text{x}\ W_1\ \text{x}\ B_2}{A_1\ \text{x}\ V_2\ \text{x}\ W_2\ \text{x}\ B_1}\ \text{x}\ 100$$

where

$A_1$ = area of standard 6-bromo-2,4-dinitroaniline;

$V_1$ = injection volume of standard 6-bromo-2,4-dinitroaniline;

$W_1$ = weight of standard 6-bromo-2,4-dinitroaniline;

$B_1$ = total volume of standard 6-bromo-2,4-dinitroaniline;

$A_2$ = area of 6-bromo-2,4-dinitroaniline peak in sample;

$V_2$ = injection volume of sample;

$W_2$ = weight of sample; and

$B_2$ = total volume of sample

## ANNEX B

[*Table 1, S1 No.* (iv)]

## DETERMINATION OF 2,4-DINITROANILINE CONTENT BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

### B-1 GENERAL

High-performance liquid chromatography or high-pressure liquid chromatography (HPLC) is a chromatographic method that is used to separate a mixture of compounds in analytical chemistry and biochemistry so as to identify, quantify or purify the individual components of the mixture.

### B-2 APPARATUS

**B-2.1 Binary Gradient Liquid Chromatography System**, capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

**B-2.2 Column**

C18 column of 100 Å with length 250 m, internal diameter 4.6 mm and particle size5 µm or equivalent.

### B-3 REAGENTS

**B-3.1 Acetonitrile**, HPLC grade.

**B-3.2 Water**, HPLC Grade.

**B-3.3 Tetra-*n*-butylammonium Hydrogen Sulphate**

**B-3.4 2,4-dinitroaniline**, known purity.

### B-4 STANDARD PREPARATION

**Solution A** — Weigh accurately 0.005 0 g standard 2,4-dinitroaniline in 100 ml volumetric flask. Dissolve it in acetonitrile and make it up to the mark with acetonitrile**.**

Take 1 ml of solution A in to 10 ml volumetric flask. Dilute it up to the mark with acetonitrile.

### B-5 SAMPLE PREPARATION

Weigh accurately 0.050 0 g sample in 100 ml volumetric flask. Dissolve it in acetonitrile and make it up to the mark with acetonitrile.

### B-6 BUFFER PREPARATION

Take 1.00 g tetra-*n*-butylammonium hydrogen sulphate in 500 ml volumetric flask. Add 200 ml HPLC grade water and complete dissolve it. Make it up to the mark with HPLC grade water.

**B-7 FLOW RATE**, 1.00 ml/min.

### B-8 MOBILE PHASE

| Acetonitrile | Buffer |
|---|---|
| 40 | 60 |

**B-9 COLUMN OVEN TEMPERATURE,** 40 °C.

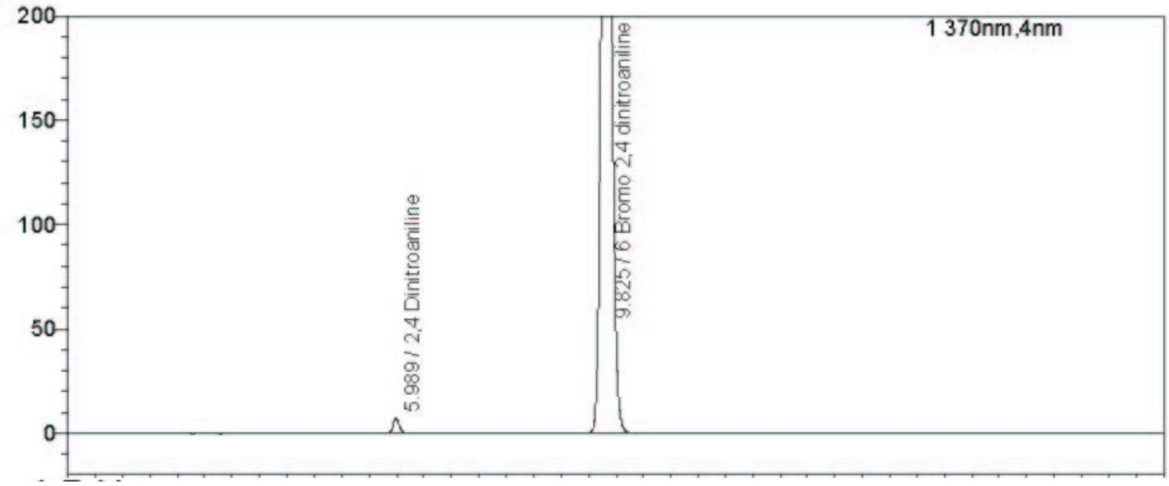
**B-10 INJECTION VOLUME,** 5 µl.

**B-11 RUN TIME**, 20 min.

**B-12 WAVELENGTH,** 370 nm.

### B-13 PEAK TIME

| | |
|---|---|
| 2,4-dinitroaniline: | 5.98 min |
| 6-bromo-2,4-dinitroaniline: | 9.28 min |



## B-14 CALCULATION

Calculate the peak area of individual constituent pertaining to 2,4-dinitroanilineon the chromatogram of the material. The concentration of the constituent may be obtained on the basis of peak area on chromatogram obtained with known amount of pure 2,4-dinitroaniline.

$$2{,}4-\text{dinitroaniline, percent} = \frac{A_2 \text{ x } V_1 \text{ x } W_1 \text{ x } B_2}{A_1 \text{ x } V_2 \text{ x } W_2 \text{ x } B_1} \text{ x } 100$$

where

$A_1$ = area of standard 2,4-dinitroaniline;

$V_1$ = injection volume of standard 2,4-dinitroaniline;

$W_1$ = weight of standard 2,4-dinitroaniline;

$B_1$ = total volume of standard 2,4-dinitroaniline;

$A_2$ = area of 2,4-dinitroaniline peak in sample;

$V_2$ = injection volume of sample;

$W_2$ = weight of sample; and

$B_2$ = total volume of sample.

## ANNEX C

(*Foreword*)

## COMMITTEE COMPOSITION

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## Amendments Issued Since Publication

| Amend No. | Date of Issue | Text Affected |
|---|---|---|
| | | |
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